

# PEEL TEST REVISITED USING NOVEL HIGH THROUGHPUT METHODS\*

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## Introduction

The peel test is one of the most common techniques to investigate the properties of pressure sensitive adhesives (PSA).<sup>1</sup> As the demand increases<sup>2</sup> for combinatorial tools to rapidly test material performance, designing a high throughput peel test is a useful improvement of this well-established technique. A simple way to achieve this purpose consists of carrying out simultaneous parallel tests (using multiple load cells), and a gradient sample or measurement conditions along the peeling direction.

A combinatorial peel test could, however, compromise the necessary statistical accuracy required for a single force measurement. The purpose of this work is to explore the possibility of using gradient multivariant samples in a peel experiment. We investigate the potential and limitations of this high throughput peel test by probing different parameters (adherent roughness and surface energy, peel rate, adhesive and backing thickness). We focus on the mechanisms which control adhesive debonding, in particular relating to the effects of the adherent surface energy on the peel force and mechanism.

## Experimental

We perform 90° peel experiments with a custom designed apparatus (Figure 1). This setup allows the measurement of both the force  $F$  (resolution is 1 mN) applied during the peel of the tape at a given rate (100  $\mu\text{m/s}$  in the experiments presented here) and images of the contact edge through the transparent adherent.

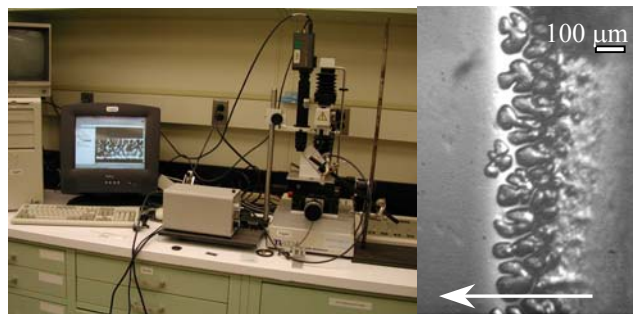


Figure 1. Experimental setup (left image) used for peel test experiments. The micrograph on the right depicts the edge morphology (peel direction is indicated by the arrow).

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We used a commercial transparent adhesive tape (Scotch 600 from 3M. Tape width  $b$  is 1.9 mm),<sup>3</sup> applied on the adherent surface at a room temperature with a commercial 2 kg (4.5 lb) roller (diameter 9.5 cm and width 4.5 cm, from ChemInstruments). The peel test was performed within a few minutes after application to the adherent.

We have investigated two model adherent surfaces. The first, which function is reference, consists of a regular glass slide (75 mm length), initially covered<sup>4</sup> with a grafted monolayer of a short alkyl silane chain of n-octadecyldimethylchlorosilane (ODS). It exhibits a homogeneous low surface energy of 26  $\text{mJ/m}^2$ . A second surface was prepared by symmetric UVO gradient exposure of the reference surface and washed with toluene, as described elsewhere.<sup>5</sup> As the exposure time increases from the edge of the sample (Figure 2, top), oxygenated functional groups are developed in the ODS layer, increasing the polarity of the surface, thus its thermodynamic energy. The gradient pattern in exposure time generates a gradient in surface energy.<sup>5</sup>

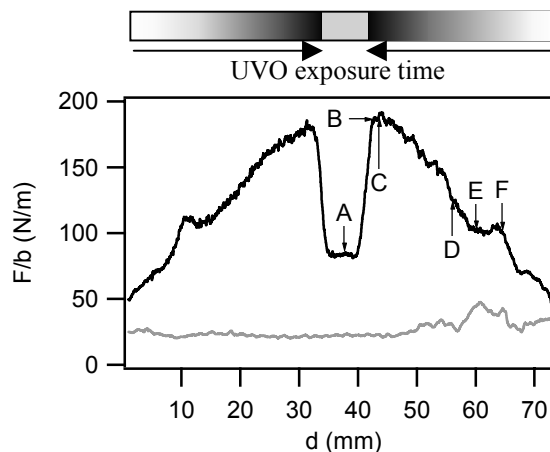


Figure 2. Evolution of the peel force  $F/b$  with the edge displacement  $d$  during the peeling of the adhesive tape from a glass slide grafted with ODS. The gray curve corresponds to a homogeneous surface, the black one to the surface with energy gradients. Top: schematic of the UV exposure time associated with the black curve.

## Results and Discussion

The evolution of the force  $F$  (divided by the tape width  $b$ ) is shown in Figure 2 for both prepared samples. In the case of a homogeneous ODS surface (gray curve), the force is constant at a value of 26  $\text{N/m} \pm 1 \text{ N/m}$  (error

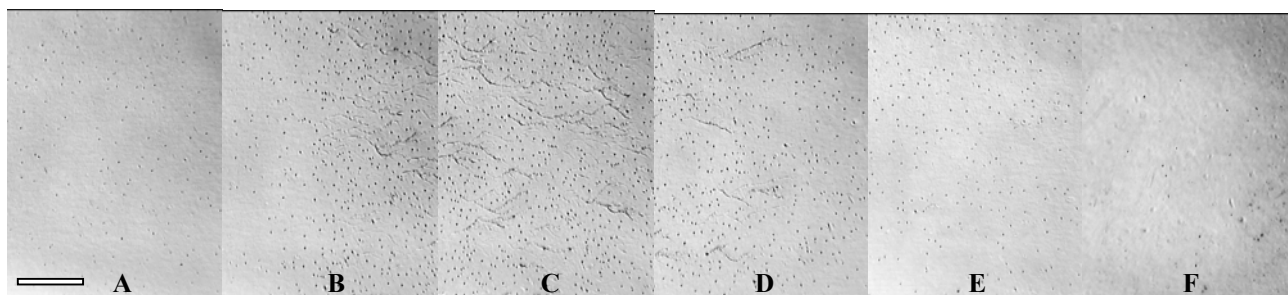


Figure 3. Optical micrographs of the adhesive tape surface after peel test (the bar on the left image represents 1 mm). The letters refers to the position shown on Figure 2. From left to right, a transition in the peel mechanism appears on B (more dot-like defects and worm-like defect appear) and on D (no change in dot-like defects, but no more worm-like defects).

bar represents one standard deviation of the data, and is taken as the uncertainty of the measurement). In the case of the gradient surface (black curve), very important variations are observed, from almost 30 N/m where the ODS layer is nearly unmodified (zero exposure time) to 190 N/m where the exposure time is the highest (40 s) and so the surface energy of the adherent is also highest (Figure 2, top). The relation between peel force and exposure time (therefore surface energy) emerges clearly: increasing the UVO exposure time increases linearly the peel force.

The symmetry of the load variation along the sample (Figure 2, black curve) also suggests that the result of the test is not sensitive to the peel direction relative to the energy gradient. This is not necessarily true in other systems or gradients including, for example, the case of a steep gradient and a thick (or stiff) adhesive layer, due to the potential variation of substrate properties within the contact edge width.

The debonding mechanisms are also affected by the adherent surface energy. Figure 3 shows micrographs of the adhesive surface, taken after the complete peel of the tape at different positions (A-F in Figure 2). An obvious morphology change occurs around the abrupt force increase at point B: from a few dot-like post-test defects in A to multiple dots and worm-like defects in C. Thereafter, as the surface energy decreases, the number of defects also decreases and the worm-like defects eventually disappear at point D. This sequence suggests a change in debonding mechanism at point D without a significant force change. Such observations demonstrate the usefulness of simultaneous imaging as a complement to the force analysis.

## Conclusion

We have demonstrated the feasibility of a high throughput peel test, using an adherent surface patterned with energy gradient. The advent of such combinatorial peel test measurements appears promising both for fast screening of adhesive properties and for fundamental studies of debonding mechanisms,<sup>6-8</sup> especially through the correlation between changes in edge morphology and load variations.

## Acknowledgements

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## References

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2. J. Carson Meredith, A. Karim and E. J. Amis, *MRS Bull*, 2002, **27**, pp 330-335.
3. Equipment and instruments or materials are identified in the paper in order to adequately specify the experimental details. Such identification does not imply recommendation by NIST, nor does it imply the materials are necessarily the best available for the purpose.
4. To graft the SAM on a glass slide, the latter is first cleaned with solvents and exposed for 30 min in a UVO cleaner. Then the glass slide is placed for 24 h under vacuum in the vicinity of a watch glass containing a few drops of the silane solution to be grafted. The glass slide is then rinsed with toluene before curing at 125 °C under vacuum for one hour.
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